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Small-angle scattering of X-rays

By Göran Wassberg and Kai Siegbahn

With 9 figures and 3 diagrams in the text

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§ 1. Introduction

By means of X-rays the size and shape of particles can be measured regardless of there being any lattice structure or not when diffraction from the particles as a whole is studied. Frequently the particles of interest are very large, considerably larger than the wavelength of the X-ray beams. The diffraction patterns will then appear at very small angles. For Cu Kα_1 (1.54 A) the radiation will be scattered less than 1° from particles of the size of 100 A. Measurements can be made down to 1′, and therefore particles of many thousand A can, at least in principle, be examined.

The technique of small-angle scattering is comparatively new. The first article on the subject was published in 1939 by Guinier (1), where he discussed the basic theories for the scattering of X-rays at small angles from small discrete particles. Since then the scattering technique has been used in a great number of fields, where the size and shape of small particles are relevant; e.g. in the study of proteins and virus, which have a very convenient size for examinations of this kind, and which, furthermore, are easy to obtain of uniform size and in sufficient quantities.

To verify the theories for small-angle scattering, the results, obtained from small-angle scattering experiments, can be compared with those obtained by other methods, esp. the methods, which give the results more directly. Usually the latter methods can be used for particles that can be seen through an electron microscope, or for crystals, the size of which can be determined from the width of the diffraction lines in a Debye–Scherrer exposure.

A good electron microscope has a resolving power of about 20 A. For particles of some hundred A, one can get relatively accurate pictures and as particles of that size are suitable for small-angle scattering, one has here a method of checking the X-ray results. Small-angle scattering and electron microscopy are two methods complementary to each other.

Electron microscopy gives direct information about the shape as well as the size of every particle, regardless of the particles being of the same size or not. The precision is often greater than in small-angle scattering measurements, provided that suitable samples can be prepared. The sample with the particles must be absolutely dry, since it goes into the vacuum of the microscope. On evaporation, the particles may change their modification in a way that cannot be predicted. For the X-ray measurements, on the other hand, the particles can be put into a solution where their proper size and shape can be measured in their correct environment. A virus in a solution has quite different characteristics than a crystallized virus.

In § 2 of this paper various types of equipment for small angle scattering will be discussed. One of the greatest problems is the long exposure time and in § 2 we shall describe the successful experiments we have made to reduce it by bending crystals spherically so that they are double-focussing. Two different methods of bending the crystals have been employed. The circular quartz crystals used have either been bent between two circular edges or been fixed to a spherically polished crystal holder by pumping vacuum between the crystal and the holder. The crystals, bent in these two ways, show different characteristics. The first method gives slightly ellipsoidally bent crystals which give perfect double focussing if the centre of the crystal is used. This type of crystal holder has been used in the equipment for the investigations of polystyrene latex which are described in § 4. Because of the good focussing and the great dispersion it has become possible to examine the scattered rays at very small angles, considerably smaller than one has been able to work with before.

In § 3 a short summary is given of the basic theory that we will make use of in this and following papers.

§ 2. Equipment

General conditions concerning the equipment

In the case of small angle scattering of X-rays one must measure low intensities near the direct beam. The ratio between the direct beam intensity and the intensity of the scattered rays is $10^{-4}-10^{-6}$. Thus great care must be taken in defining the incident beam and screening the parasitic beam that may strike the detector within the small angle area. The beam should be monochromatic and the intensity as high as possible to reduce the time of exposure or measurement to reasonable values.

As long a wave-length as possible should be used to get a better dispersion and to reach large periodicities, but with a longer wave-length the experimental difficulties will increase.

The Cu Kα_1 radiation (1.54 A) is that most frequently used in this connection. Rays of this energy can pass through air without loosing too much of their intensity by air absorption. 8.3 A (Al Kα) would be a much more suitable wave-length. The equipment does not necessarily have to be kept in vacuum but can be in hydrogen atmosphere. Even at 8.3 A the absorption of X-rays in aqueous vapour is not greater than that saturated vapour of low pressure can be kept in the equipment. This is of great importance if the sample is to be kept at a constant concentration in order not to change its character during the exposure.

Previous constructions

Various pieces of equipment have previously been constructed, the main types of which will be discussed below.

In one type of instrument the beams from the X-ray tube are defined geometrically by slits or pin-holes. If very narrow slits and a large distance between the X-ray source and the plate are used, a very high resolving power can be obtained and scattering angles down to less than 2′ (1500 A for Cu Kα) can be measured. A great disadvantage is that the intensity decreases with increasing resolving power and the exposure time becomes too long for materials of low scattering power.

A monochromatic beam is produced by filtering away the Kβ component. The filtered ray is not strictly monochromatic which makes the measurements uncertain.

A strictly monochromatic beam is produced by crystal reflexion. The best instruments of this kind are those with one or two cylindrically bent crystals as monochromators. The beams converge to a very sharp line focus and the intensity is very high. The instruments with one crystal are not suitable for angles smaller than 40′ (100 A for Cu Kα) because of the diffusely scattered radiation from the crystal.

To reach smaller angles two bent crystals can be used to reduce the parasitic scattering. (2)

The slit screens off all parasitic scattering and the second crystal is only struck by the monochromatic beam. The diffuse scattering from the second crystal is very small and therefore one can measure down to 10′ (300 to 400 A for Cu Kα). The loss of intensity by reflexion in the second crystal is partly compensated by the fact that considerably larger solid angle can be used (1° or larger at the final beam).

The disadvantage with all the systems, where one obtains line-shaped images of the direct beam, is that the uncertainty in angle is great at small angles. If this uncertainty is reduced by using a low slit height, the intensity is also reduced.

The ideal set-up would be a system where beams could be focussed to a point instead of a line. Only a few experiments have been made to produce point focussing monochromators.

The first intention was to bend a crystal along a toroidal surface, which, theoretically, would give a point focus. These experiments were not successful. Either

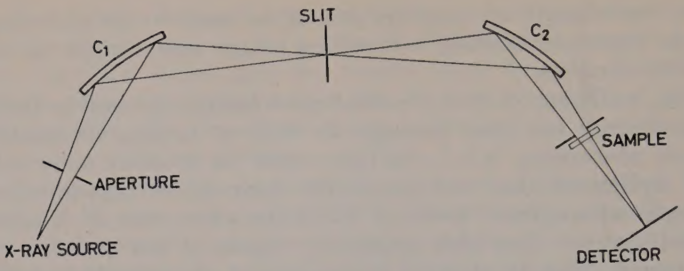

Fig. 1. Set-up with two cylindrically bent crystals to reduce the diffuse scattering.

the crystals broke or the lattice structure was damaged so that good focussing could not be obtained. A toroidal crystal of aluminium has been prepared by Hägg and Karlsson (3). They obtained a beam 50 times as intense as that from a cylindrically bent quartz crystal. This monochromator has not been used in small-angle scattering experiments.

Du Mond (4) has shown that one can get a good point focus by reflexion in two cylindrically bent crystals placed after each other in such a way that they focus the beam in two perpendicular directions.

The intensity losses at this double reflexion are great because of the polarization of the beam after the first reflexion. At a Bragg angle of 45° the polarization is total and no radiation is reflected from the second crystal if the latter has the same Bragg angle. Du Mond's crystals have a Bragg angle of 40°, and therefore the losses were unreasonably great.

To avoid the effect of polarization, large or small Bragg angles must be used.

A point focussing monochromator with one crystal has been constructed by D. W. Berreman (5). The crystal is cut out from a quartz block in such a way that when the crystal is bent cylindrically the atomic planes are along a toroidal surface. The intensity is very good. The crystal reflects almost 3 % of the incident Cu Kα radiation to a convergent beam, one meter long. The smallest measurable angle is 20′ (200 A for Cu Kα).

Our equipment and its characteristics

At large Bragg angles a spherical surface will closely approximate the toroidal surface, which would give a point focus. In order to examine the possibilities of bending a quartz crystal spherically, we have made a series of experiments.

The crystal is circular with a diameter of 60 mm and a thickness of 0.5 mm. It is cut along the atomic planes $1 0 \bar{1} 0$ and has a lattice constant of 4246 XU. This gives Bragg angles in the vicinity of 80° for Al Kα in the first order, for Ag Lα_1 in the second order and for Cr Kβ_1 and Mn Kα_1 in the fourth order. The wavelengths are 8.3 A, 4.15 A, 2.08 A and 2.10 A respectively.

The crystals have been bent in crystal holders of two different types. In one of them the crystal has been bent between two carefully polished circular edges to a radius of curvature of 1 meter. Fig. 2 shows a sketch of the crystal holder and fig. 3 a photograph.

In the experiments we used a point focus tube manufactured by Hilger. The focal spot was only 40 μ in diameter and the power of the tube was 5 W with

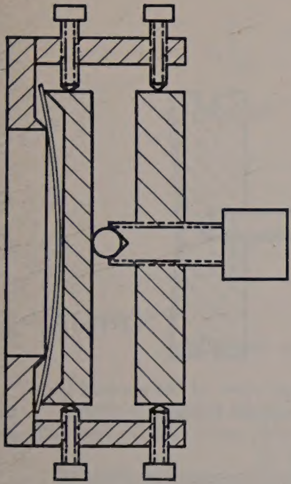

Fig. 2. Schematic drawing of the crystal holder.

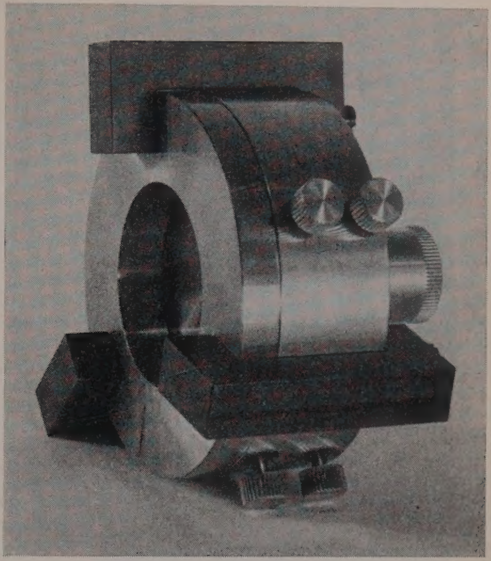

Fig. 3. Photograph of the crystal holder.

an anticathode of chromium, the β_1-line of which has a suitable wave-length. It appeared that the center of the crystal within a diameter of about 15 mm could be used. The parts of the crystal lying nearer the edges had a somewhat different radius of curvature. The crystal surface in the middle, which was usable, was not exactly spherical but slightly ellipsoidal, due to the crystal's elastic properties. This, however, could easily be used to reduce the astigmatic aberration, which always appears when a point not situated on the optical axis is reproduced in a spherical mirror. The incident and reflected beams must then lie in the same plane as the major axis of the ellipsoid. Fig. 4 shows a photograph of the focussed beam. One can see that the focussing is extraordinarily good and that the astigmatic aberration has been completely eliminated. This type of crystal holder has been used in the small-angle scattering experiments, which will be described further on in this paper.

Fig. 5 shows the other type of crystal holder. It is made of tempered steel and its surface is polished spherical with an accuracy of $\pm$ 1 light wave-length. The radius of curvature is 1 meter. When vacuum is pumped under the crystal it sticks to the surface of the holder to such an extent that no light interference appears, except along the outermost 3–4 mm of the crystal. Thus about 50 mm of the crystal surface can be used. If the crystal is to stick to the crystal holder, air must not ooze in at the crystal edge. This is prevented by packing araldite along the edge. The most troublesome problem has been to keep the crystal quite free from dust.

A speck of dust smaller than one light wave-length manifests itself by the interference patterns around the speck and the result is a diffuse X-ray diffraction pattern. However, by means of an antistatic cloth it has been possible to remove all the dust from the crystal.

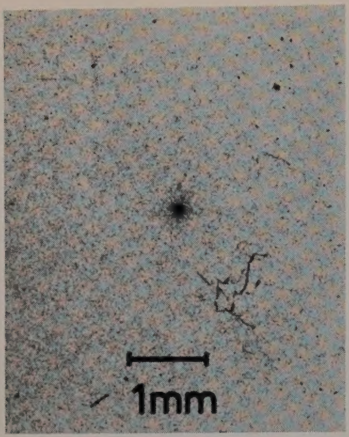

Fig. 4. Photograph (magnified 11 times) of the point focussed X-ray beam.

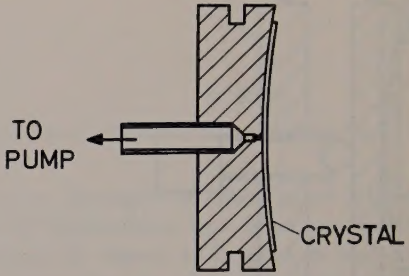

Fig. 5. Schematic view of the second type of crystal holder.

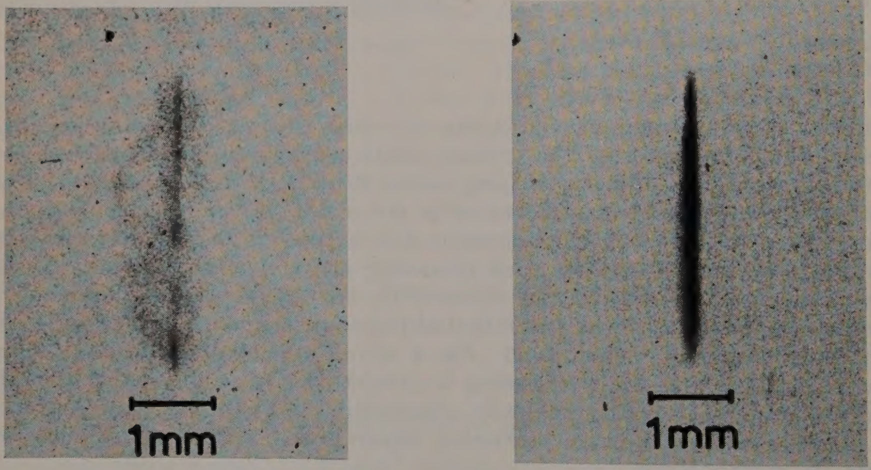

Fig. 6. Photograph (magnified 11 times) from exposures made with the crystal holder shown on fig. 5. The photograph to the left shows the diffuse diffraction pattern which results when the crystal is not quite free from dust.

As the crystal surface is not quite spherical one will get (because of the astigmatic aberration) not one point focus but two line foci F_1 and F_2 in fig. 7, which become larger the larger the part of the crystal is that is being used.

By using two equal spherical crystals arranged as shown in fig. 7 an anastigmatic system can be obtained and point focussing is achieved.

If two slits are placed at F_1 and F_2 so that the parasitic scattering is eliminated, the other crystal will be exposed to the monochromatic beam only. The diffuse scattering from the second crystal will become less. (Cf. the monochromator with two cylindrical crystals.)

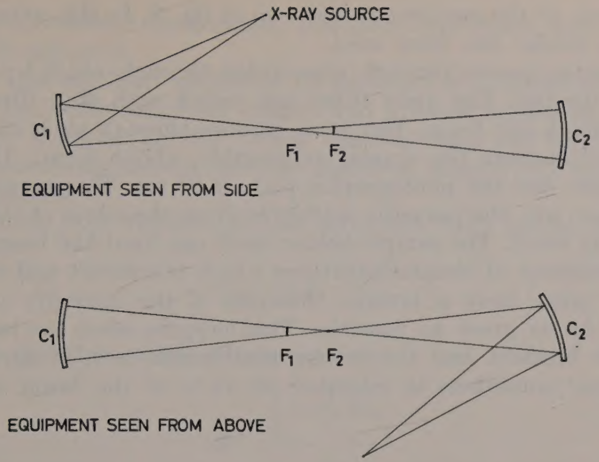

Fig. 7. Schematic view of the point focussing monochromator with two spherically bent crystals. The sample has been placed in the convergent beam along C_2. The parasitic scattering from C_1 is eliminated by two slits at F_1 and F_2.

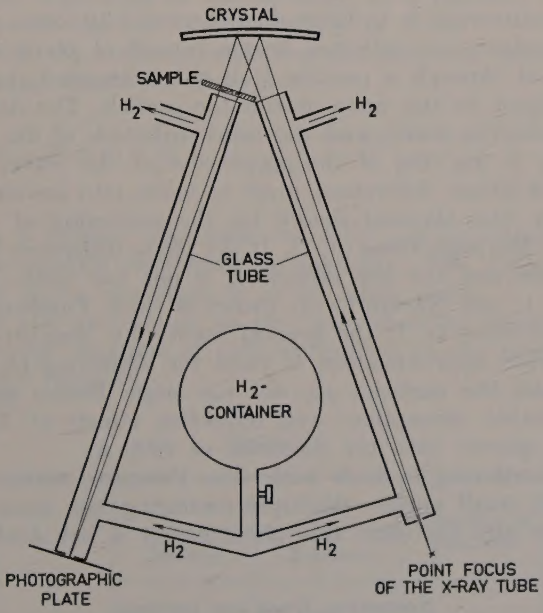

Fig. 8. Experimental set-up for investigations of small angle scattering from polystyrene latex.

As the Bragg angle is in the vicinity of 80° the polarization losses will be insignificant. The reflexion power of a crystal is greatest at large Bragg angles (proportional to the tangent of the Bragg angle), and therefore a very high intensity can be expected with this equipment.

Diffraction patterns for a sample in the form of powder have been investigated.

The arrangement of the equipment is shown in fig. 8. In this experiment the first type of crystal holder has been used.

The X-ray beam passes through glass tubes through which hydrogen gas from a container is flowing. The glass tubes are sealed with thin films which absorb very little of the X-ray beam. The beam passage through air is about 15 cm. The sample is placed as near the crystal as possible, about 5 cm. The distance between the sample and the photographic plate is 95 cm. This causes the dispersion to be very great and the parasitic scattering from the edges of the sample holder to be exceedingly small. The sample holder itself can limit the beam. Thus one can avoid all adjustments of circular apertures which is difficult and time-consuming.

The sample must have a certain thickness if the intensity of the scattered radiation is to be as great as possible. This happens when the ratio between the intensity of the incident and the transmitted beams is *e*. If deviations of about 5 % from optimal conditions is tolerated 50–75 % of the beam will be absorbed in the sample.

§ 3. Theory

General assumptions

If small angle scattering is to be studied theoretically, some assumptions must be made. The incident and reflected beams consist of plane waves. When the beam is transmitted through a particle it is not attenuated and does not change its phase with respect to the wave outside the particle. The electrons in the particle scatter the incident wave, and the total amplitude of the wave scattered in a certain direction is the sum of the amplitudes of the wave, scattered at each electron, where the phase differences must be taken into account. These assumptions are made in the classical theory for the scattering of X-rays. It is the approximation by Rayleigh–Gans (6, 7). If the phase difference between the wave through the particle and the one outside it is not too small, but the index of refraction is near 1, von Nardroff's (8) theory is valid. Furthermore, if the index of refraction is not close to 1, the general theory by Mies (9) must be used.

The Rayleigh–Gans approximation is valid for scattering of X-rays as long as the wave-length and the particles are not too large. Henke and Du Mond (10) have found noticeable absorption- and refraction effects at N Kα (31.6 A) for polystyrene latex spheres with the diameter of 2800 A.

Only coherent scattering is dealt with. The Compton scattering is ignored, as it is very small at small angles. (Multiple scattering has some influence only in a few special cases and therefore this phenomenon is not dealt with.)

Scattering from one particle

On calculating the scattering from one particle, a molecule or a group of atoms, one finds that the intensity of the scattered beam is dependent on the shape of the particle. This dependence is given by the scattering function $i(h)$ of the particle where

$$h = \frac{4\pi \sin \vartheta}{\lambda} \tag{1}$$

$2\,\vartheta$ is the angle between the incident and scattered beam and λ is the wavelength of the X-radiation.

The function $i(h)$ has been calculated for particles of different shape. Guinier (1) has calculated the scattering from spheres and rotation ellipsoides with a uniform electron density, Leonard (11) for hollow spheres, Oster and Riley (12) for hollow spheres and spheres with radially periodic densities, Kratky (13) and Porod (14) for cylinders and prisms.

For small values of h Guinier (1) has derived a very useful expression

$$i(h) = \exp - \frac{h^2 R^2}{3} \tag{2}$$

where R is the gyration radius of the particle

$$R^2 = \frac{\int\limits_v \varrho_m r_m^2 \, dv_m}{n} \tag{3}$$

r_m is the radius vector from the origin to the m:th volume element, where ϱ_m is the charge density. n is the number of electrons in the particle.

If one plots the logarithm of the intensity as a function of h^2 one gets R^2 has from the slope of the line in the vicinity of $h=0$.

For spheres with the radius a and constant electron density Rayleigh (15) derived the following expression for $i(h)$:

$$i(h) = \Phi^2(h\,a) = \left[3\,\frac{\sin h\,a - h\,a \cos h\,a}{h^3\,a^3}\right]^2 = \frac{9\,\pi}{2}\left[\frac{J_{\frac{3}{2}}(h\,a)}{(h\,a)^{\frac{3}{2}}}\right]^2 \tag{4}$$

This function has a central maximum for $h=0$ and is followed by a series of maxima, which is rapidly decreasing in intensity. The distance between two maxima expressed in $h\,a$ converges to π for large angles.

The maxima of $i(h)$ correspond to the case when the Bessel function of order 3/2 vanishes and the maxima of $i(h)$ correspond to the case when the Bessel function of order 5/2 vanishes.

Table 1.

	$h\,a$		Intensity of maxima
	Minima	Maxima	
0		0	1
1	4.493	5.765	0.00742
2	7.725	9.095	0.00127
3	10.90	12.32	0.00039
4	14.07	15.52	0.000154
5	17.22	18.69	0.000073
6	20.37	21.85	0.000039
7	23.52	25.01	0.0000228
8	26.67	28.17	0.0000142

Scattering from a great number of particles

So far only the scattering from one particle has been dealth with. In all the experiments the radiation is scattered from a sample, consisting of a great number of particles. One is interested in the conditions under which one can use the results of one-particle scattering and what modifications must be made because of the interaction between the particles.

If the particles are identical and if there is no interaction between the particles the intensity of the scattered beam is the sum of the intensities scattered from each particle. $I(h) = N\,i(h)$ where N is the number of scattering particles. This is valid for a gas or for particles in a very dilute solution where the particles have a random distribution.

If the concentration of the solution is increased this is not valid. The diffraction patterns change because of interference between waves scattered by neighbouring particles and some corrections must be made. This problem has been dealt with by Menche (16) and Fournet (17) for particles of arbitrary shape.

Especially for spherical particles calculations have been made by Gingrich and Warren (18) and Yudowitch (19). They obtained

$$I(h) = N\,\Phi^2(ha)\left\{1 + P\left[5\,\frac{\sin 2ha}{2ha} - 6\,\Phi(2ha)\right]\right\} \tag{5}$$

(cf. equation 4).

P is a parameter and a measure of how closely packed the particles are. P is equal to the ratio between the number of particles in a volume and the maximum number of particles that would be in the same volume

$$0 \leqslant P \leqslant 1$$

$P = 0$ is equivalent to an infinitely diluted solution. $P = 1$ for a facecentered cubic arrangement of the particles.

By considering the sample as a fluid of hard spheres we get a thermodynamic equation (17)

$$I(h) = N\,\Phi^2(ha)\,\frac{1}{1 + \frac{8v_0}{v_1}\,\varepsilon\,\Phi(2ha)} \tag{6}$$

$$0 < \frac{8v_0}{v_1}\,\varepsilon < 6$$

v_0 is the volume of the particle, v_1 is the mean volume available for each particle, ε is a constant which is approximately equal to 1.

For very diluted solutions, equations (5) and (6) change into equation (4).

Increased concentration generally causes the intensity to decrease at the smallest angles while only small intensity changes take place at larger angles.

If one uses Guinier's approximation, equation (2), to find the gyration radius, one gets a value that is too low because of the particle interaction. If a series of measurements are made at different concentrations, one can get the real gyration radius.

At high concentrations or strong particle interactions there sometimes appears a more or less diffuse interference maximum which is indicated in equations (5) and (6).

§ 4. Examination of polystyrene latex

Polystyrene latex from the Dow Chemical Co., USA, with the designation 15 N-7 has been examined (20). This sample consists of spheres of uniform size with a stated diameter of 3400 A and with a mean deviation of 52 A. The values have been obtained from electron microscopic measurements.

The preparation of the latex was rather easy. The sample holder consisted of a metal plate, 0.5 mm thick, in which a hole with the diameter 10 mm had been bored. A thin film was fastened on one side of the hole and latex soaked in water was evaporated onto the film until a suitable sample thickness was obtained. Then a thin film was fastened over the other side of the hole.

As the latex particles are spheres of uniform size a great effect of the interaction between the particles can be expected.

The intensity distribution for scattering without any interaction is given by equation (4), which has been plotted for $h a < 10$, diagram 1. Table 2 gives the values of $i(h) = \Phi^2(h a)$ for different values of $h a$.

Table 2 a.

$h a$	$i(h)$
0	1
0.5	0.951
1.0	0.816
1.5	0.628
2.0	0.427
2.5	0.249
3.0	0.119
3.5	0.0419
4.0	0.00758
4.5	0.00000

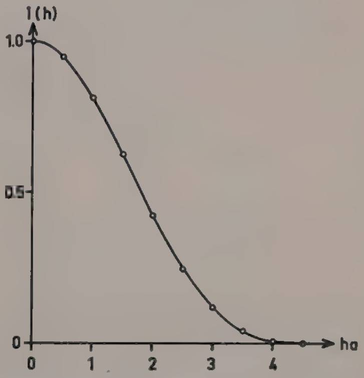

Diagram 1 a. The value of $i(h) = \Phi^2(h a)$ for $0 \leqslant h a \leqslant 4.5$.

Table 2 b.

$h a$	$i(h)$
4.5	0.00000
5.0	0.00326
5.5	0.00689
6.0	0.00704
6.5	0.00449
7.0	0.00163
7.5	0.000140
8.0	0.000159
8.5	0.000835
9.0	0.00126
9.5	0.00108
10.0	0.000554

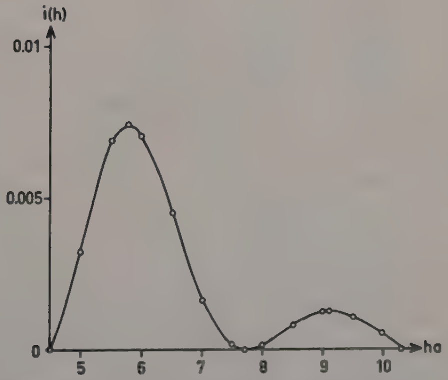

Diagram 1 b. The value of $i(h) = \Phi^2(h a)$ for $4.5 \leqslant h a \leqslant 10$.

Table 3. Summary of the values of $h\,a$ when $i(h)$ has its maximum at $P=0$, $P=\frac{1}{2}$ resp. $P=1$.

Max no.	$P=0$	$P=\frac{1}{2}$	$P=1$
0	0	2.5	2.85
1	5.765	5.99	6.18
2	9.095	9.23	9.36
3	12.32	12.44	12.52
4	15.52	15.60	15.67
5	18.69	18.76	18.82
6	21.85	21.91	21.96

The effect of the interaction is given by equations (5) and (6). In equation (5) the values $P=\frac{1}{2}$ and $P=1$ have been substituted, and the maximum values of $i(h)$ have been calculated. The results of these calculations have been summarized in table 3.

Equation (6) has been used to calculate the intensity distribution for $h\,a<3.5$. As parameters we have chosen $\frac{8\,v_0}{v_1}\varepsilon=2.4$ and 6. These results are given in diagram 2 and tables 4 and 5. This should be compared with the curve obtained when interaction is neglected $\frac{8\,v_0}{v_1}\varepsilon=0$ in diagram 1.

Table 4.

$h\,a$	$i(h)$		
	$\frac{8\,v_0}{v_1}\varepsilon=2$	$\frac{8\,v_0}{v_1}\varepsilon=4$	$\frac{8\,v_0}{v_1}\varepsilon=6$
0	0.3333	0.2000	0.1429
0.5	0.3389	0.2062	0.1482
1.0	0.3538	0.2258	0.1658
1.5	0.3716	0.2639	0.2050
2.0	0.3636	0.3166	0.2805
2.25	0.3332	0.3332	0.3332
2.5	0.2811	0.3227	0.3788
2.75	0.2133	0.2663	0.3544
3.0	0.1430	0.1791	0.2396

Table 5. Values of $h\,a$ at $i(h)_{\max}$ from diagram 2.

$\frac{8\,v_0}{v_1}\varepsilon$	Values of $h\,a$ at $i(h)_{\max}$
2	1.6
4	2.3
6	2.5

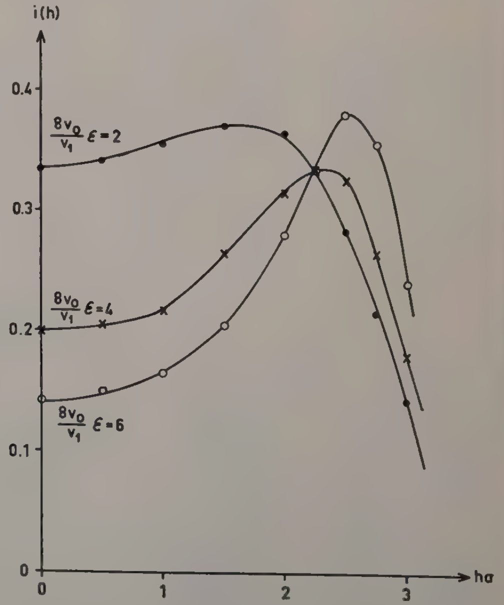

Diagram 2. The value of $i(h)=\Phi^2(h\,a)$ for $0\leqslant h\,a\leqslant 3.0$ according to equation (6) for different values of the parameter $\frac{8\,v_0}{v_1}\varepsilon$.

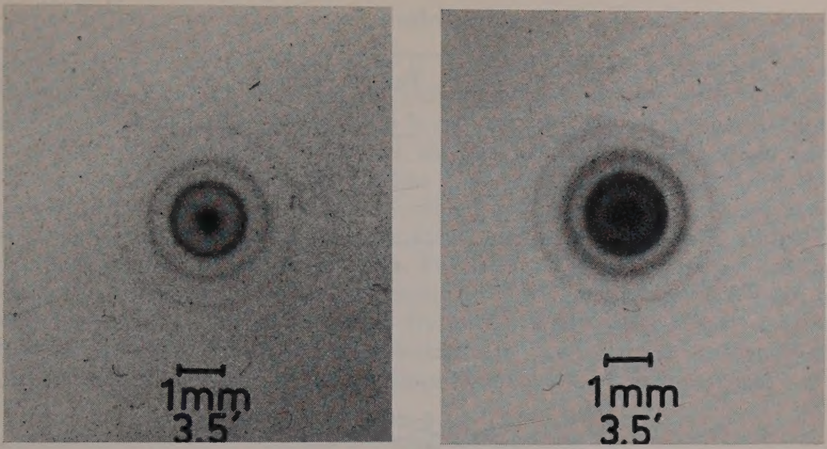

Fig. 9. A photograph (enlarged about six times) of two of the negatives.

Table 6. The diameters of the different interference maxima measured in a comparator.

Ring no.	Diameter in mm
0	1.385
1	2.41
2	3.605
3	4.77
4	5.96
5	7.175
6	8.395

Table 7. The maximal error $< 1\%$.

Max no.	Particle diameter in A for		
	$P=0$	$P=\frac{1}{2}$	$P=1$
0	—	2281	2601
1	3023	3142	3241
2	3189	3236	3282
3	3265	3296	3317
4	3291	3308	3324
5	3292	3305	3317
6	3289	3297	3305

A number of exposures with different exposure times have been made. The time of exposure varied between 3 minutes which was enough to give the zero maximum and 40 hours when 7 maxima were obtained.

The power of the X-ray tube was 5 W (25 kV − 200 μA) and the Cr Kβ_1-line ($\lambda = 2.0806$ A) is used. The registration was made with Ilford Industrial Type G X-ray film, which was developed in the Ilford developer I D–42.

Table 8.

$\frac{8 v_0}{v_1} \varepsilon$	Particle diameter in A
2	1460
4	2099
6	2281

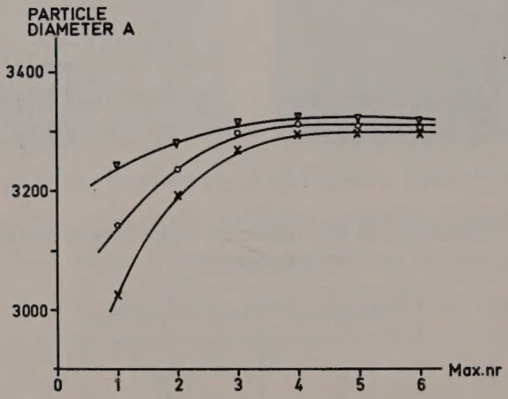

Diagram 3. Particle diameter as a function of the number of the maximum for $P=0$, $P=\frac{1}{2}$ and $P=1$.

The result according to equations (5) is given in table 7.

Equation (6) has only been used for the zero maximum, since it turned out that the correction for the other maxima was too small to give constant values for the diameters of the particles.

Diagram 3 shows the diameters of the particles as a function of the number of the maximum. (According to table 6.)

For the zero maximum neither equation (5) nor (6) give a correct value for the particle diameter for any permitted parameter value. Moreover, equation (5) gives negative intensity for $P=1$ within the zero maximum. These results, as well as those in diagram 3, indicate some effect, which arises at very small angles and which has been neglected in the theoretical treatment of the problem. The scattering angle 2ϑ is only 2.5′ for the zero maximum.

The reason for this effect might be found if a series of experiments were performed with latex solutions of various concentrations. By varying the sample thickness, it would be possible to discover a prospective influence from multiple scattering. Furthermore, one would have to make a series of experiments to examine the kind of parameters, which could have some influence on the ordering of the particles during their evaporation.

Previous measurements have been made by Du Mond (4) and Yudowitch (21). They have only published measurements of the outer maxima (Du Mond from no. 4) and have not made any examinations of the conditions at the smallest angles.

§ 5. Conclusions

We have shown in this paper that it is possible by our methods to achieve two-dimensional focusing of X-rays, resulting in a sharp point focus of the monochromatic beam. This point focus is remarkably free from undesirable blackening of the photographic plate and the conditions for small angle scattering studies are thus very favourable. It has been possible to bring down the times of exposure appreciably. Furthermore, the great dispersion of the arrangement and the sharp definition of the point focus makes it feasible to study the diffraction patterns at very small angles. Particles of large dimensions are therefore accessible for closer analysis. Latex particles of diameters up to 3400 A have so far been investigated and a deviation from theory has been observed at the first few diffraction rings. A new complete instrument is now under construction which will incorporate a number of design improvements for routine work.

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